

Name I.C. LEWIS / T. PARRO
Notebook Number 195 - 129
Subject Chemistry of Building Materials, New Products + Experiments

Dates From _____ To _____

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Parma Ohio 44130

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52

Subject Initial Data of c/c Composite via BP Process
Cross-Reference (if any)

(c/c comp BP)

Purpose:

To obtain initial wt and dimension prior to vacuum impregnation w/
 a "T-143 TYPE" resin.

Mat'l:

c/c composite via BP Process # 4-1 : Revd from P. S. rocky 4/30/01. From
 1st Lawrenceburg Trial. Block 4-1 ~1.5 x 3.0 x 7.0 inches. From 4th block in
 the series. Made w/ 0.25" long pitch fibers and keilley 155 p. beth.
 Wool Ratio 85/15 + 5 with sulfur based on the pitch weight.

Procedure:

Ultrasonic washed in deionized H₂O 3x, 5 min intervals \Rightarrow Vacuum
 dried overnight at $\sim 150^{\circ}\text{C}$ to 2.4 mm pressure.

Unloaded & cooled in desiccator. 5/11/01. Weighted & dimensioned
 5/11/01 \Rightarrow loaded into oven at $\sim 150^{\circ}\text{C}$, 5.0 scfm oxygen purged until
 loaded into VI unit.

Weight & Dimension: (5/11/01)

wt (g)	L ₁	L ₂	L ₃	Ave LEN.	w ₁	w ₂	w ₃	Ave width	H ₁	H ₂	H ₃	Ave ht.	VOL. (cc)
1139.84	191.91	192.27	192.62	192.07	83.78	83.36	82.32	83.15	47.21	46.71	46.65	46.81	748.36

$$\Rightarrow \text{Density} = 1.522 \text{ g/cc}$$

Comments:

composite contains severe defects, such as fissures and splits. One
 area appears to contain iron oxide.

VI and Cure: Ref. 195-123-~~555~~ 55256

Performed and Recorded by:

Directed by: J. Lin

Read and Understood by:

Date / /

Date / /

Date / /

Subject Preparation of ② 900 ml Aliquots of 50/50 by Volume GP-5432/Furfural (click center) 53
Cross-Reference (if any)

Purpose:

To impregnate 195-129-52 w/a "T-143 Type" resin.

Materials:

GP-5432: Lot # 19588. Rec'd from Georgia-Pacific 9/28/96. LIMS # A96-03635. Stored in Freezer. Ass't Mod MCC (2) = 48.6 ($\sigma = 0.37, n=3$), Brookfield Viscosity = 157.3 cPs at 71.0°F, DSC Data: Ref. 195-105-49, TGA Yield (900E) = 47.5%. Current Visc = 283.5 cPs at 68.7°F. Furfural: Reagent Grade (Fisher). Rec'd 1/15/01. 2nd Aliquot used 60 ml of ② 500 ml rec'd 5/15/01 \Rightarrow Balance = 940 ml.

Preparation 1st Aliquot: (5/14/01) - 1L Erlenmeyer Flask, 450 ml GP-5432 + 450 ml Furfural

$$\begin{aligned} \text{FLASK(etc)} + \text{GP-5432}_{\text{wt}} &= 996.3 & \text{FLASK(etc)} + \text{FURFURAL}_{\text{wt}} &= 1516.0 \\ \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{455.9}} & \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{996.3}} \\ 450 \text{ ml } \text{GP-5432}_{\text{wt}} &= 540.4 \text{ g} & 450 \text{ ml } \text{FURFURAL}_{\text{wt}} &= 519.7 \text{ g} \Rightarrow 50.98\% \text{ GP-5432 by wt} \end{aligned}$$

Comments:

May stirred w/o external heat for 10 min after combining \Rightarrow Transfer to 32 oz glass jar.

Characterization of the 1st Aliquot: (5/15/01)

Brookfield (LVT) RT Viscosity:

$$\text{Viscosity} = \underline{\underline{18.3}} \text{ cPs at } \underline{\underline{71.0}}^{\circ}\text{F} \quad \text{Spiral #1, 60 RPM, Factor = 1}$$

Spec. Grav. by RT:

$$\text{S.G.} = \underline{\underline{1.190}} \text{ at } \underline{\underline{71.8}}^{\circ}\text{F}$$

Preparation of 2nd Aliquot: (5/15/01) - 1L Erlenmeyer Flask, 450 ml GP-5432, 450 ml Furfural

$$\begin{aligned} \text{FLASK(etc)} + \text{GP-5432}_{\text{wt}} &\approx 995.0 & \text{FLASK(etc)} + \text{FURFURAL}_{\text{wt}} &= 1512.6 \\ \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{455.9}} & \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{995.0}} \\ \text{GP-5432}_{\text{wt}} &= 539.1 \text{ g} & \text{FURFURAL}_{\text{wt}} &= 517.6 \text{ g} \Rightarrow 51.02\% \text{ GP-5432 by wt} \end{aligned}$$

Solutions combined 5/16/01. Label as 195-129-53 1st use: Ref. 195-129-54

Mod MCC of Aliquot #1: Ref. 195-129-54

Performed and Recorded by:

Date

Date

Date

Directed by:

Read and Understood by:

54

Subject, modMCC at 195-129-53, 1st Aliquot
Cross-Reference (if any)

(C/C COMPARISON)

Material:

195-129-53, 1st Aliquot : 50/50 by volume, 51.0/84.0 by weight GP-5432/Turp
 Prep. 5/14/01, Visc_{25°C} = 18.3 cPs at 31.6°F, SG_{40°C} = 1.190 at 71

modMCC determinations : (5/15/01) - ~1.5g sample size. Cured 1hr at 144°C, 0.03074

$$\begin{aligned} 1) \text{CAVC+CHIPS+SAMPLE}_{(1)} &= 18.1507 \quad \text{CAVC+CHIPS+SAMPLE}_{(144)} = 18.3297 \Rightarrow \text{Yield}_{(144)} = 50.5\% \\ \text{CAVC+CHIPS}_{(1)} &= 17.6308 \quad \text{CAVC+CHIPS+SAMPLE}_{(\text{mcc})} = 18.1502 \Rightarrow \text{Yield}_{(\text{mcc})} = 67.5\% \\ \text{SAMPLE}_{(1)} &= 1.5129g \end{aligned}$$

$$\text{TOTAL YIELD} = (0.5059 \times 0.6755) \times 100 = \underline{\underline{34.17\%}}$$

$$\begin{aligned} 2) \text{CAVC+CHIPS+SAMPLE}_{(1)} &= 18.9635 \quad \text{CAVC+CHIPS+SAMPLE}_{(144)} = 18.2000g \Rightarrow \text{Yield}_{(144)} = 49.20\% \\ \text{CAVC+CHIPS}_{(1)} &= 17.4606 \quad \text{CAVC+CHIPS+SAMPLE}_{(\text{mcc})} = 17.9770g \Rightarrow \text{Yield}_{(\text{mcc})} = 69.84\% \\ \text{SAMPLE}_{(1)} &= 1.5029g \end{aligned}$$

$$\text{TOTAL YIELD} = (0.4920 \times 0.6984) \times 100 = \underline{\underline{34.36\%}}$$

$$\begin{aligned} 3) \text{CAVC+CHIPS+5,4MPG}_{(1)} &= 18.3929 \quad \text{CAVC+CHIPS+SAMPLE}_{(\text{mcc})} = 17.6460g \Rightarrow \text{Yield}_{(144)} = 50.5\% \\ \text{CAVC+CHIPS}_{(1)} &= 16.8837 \quad \text{CAVC+CHIPS+SAMPLE}_{(\text{mcc})} = 17.1407g \Rightarrow \text{Yield}_{(\text{mcc})} = 68.6\% \\ \text{SAMPLE}_{(1)} &= 1.5092g \end{aligned}$$

$$\text{TOTAL YIELD} = (0.5051 \times 0.6869) \times 100 = \underline{\underline{34.69\%}}$$

Comments:

All samples were hard at temperature after cure at ~144°C.

$$\text{Ave Yield}_{(144)} = 50.1\%, \sigma = 0.78, n = 3$$

$$\text{Ave Yield}_{(\text{mcc})} = 68.7\%, \sigma = 1.15, n = 3$$

$$\text{Ave ModMCC} = 34.4\%, \sigma = 0.26, n = 3$$

Performed and Recorded by:**Directed by:****Read and Understood by:**
Date:**Date:****Date:**

Subject VI + Cure to 200°C of 195-129-52 #4-1 w/195-129-53
Cross-Reference (if any)

(clc composite) 55

Purpose:

To densify clc composite w/ a phenolic resin/furfural blend. To verify max vol% pitch w/ pitches in the PSC.

Materials:

- 1) clc composite: 195-129-52 #4-1 (clc composite via BP process). From 4" block in the 1st Lawrenceburg Trial. 0.25" long pitch fibers + Reilly 105 Pitch. Load ration 85/15 + 5wt% sulfur based on the pitch wt.) Wt. co., $\approx 1138.84\text{g}$, Vol co., 748.383cc , Dens., $1.5225/\text{cc}$
- 2) Impregnant: 195-129-53 (50/50 by volume GP-5432/Furfural, Prep. 5/14+15/01.
 $V_{13}(x) = (7.4)\text{ccs at } 74.5^\circ\text{F}, S.G.(51 \pm 1.188) \text{ at } 74.5^\circ\text{F}$

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15+16

Pump-down Data: (5/14-16/01)

DATE	TIME	PRESS (mTorr)	Comments
5/14	13:30	15	load clc composite fram over (i.e. ~150°C, Atm. Pressure).
"	13:40	4	
"	16:05	16	
5/15	7:20	14	
"	16:00	16	
5/16	7:25	13	
"	8:05	15	charge traps w/ dry ice-acetone
"	8:50	10	LDR
"	11:45	10	Begin VT

Impregnation Data: (5/16+17/01) - LDR w/traps charged

LDR: Initial = 10 millitorr $V_{13}(x) = 17.4\text{ccs at } 74.5^\circ\text{F}$ Drop Time = 11:45 (10 mTorr)
5min = 21 " $S.G.(51 \pm 1.188) \text{ at } 74.5^\circ\text{F}$ Onload Time = 8:45 (5/17/01)
10min = 27 " Held at atmospheric pressure for
15min = 34 " ~21 We.

Comments:

570 ml of impregnant in the 500ml cylinder funnel.

cont'd next page

Performed and Recorded by:

Directed by: J. Caw

Read and Understood by:

Date , ,

Date , ,

Date , ,

Subject
VI and Cure (was 0°C) Data of 195-129-52 #4-1 w/195-129-53
Cross-Reference (if any)

(C/L COMP RT)

Post Impregnation Data: (5/17/01)

$$W_{C/PV-1} = 1320.11g \Rightarrow W + Pickup = \underline{\underline{181.27g}} \Rightarrow W/10 Pickup = \underline{\underline{15.92}}, W/10 Pickup f \underline{\underline{20.40}}$$

$$\text{At Pan + Screen } \underline{\underline{181.4g}}$$

Curing Data: (5/17/01) - Cure at small (1 A) Pan w/ an ss. screen to determine amount of run-out.

TIME	EVEN SET	EVEN TEAR	Comments
* 8:45	48	158	Load into oven. Purge w/ argon at 5.0 SCFH (412)
9:15	"	156	Wet resin on all visible surfaces. Condensation on oven clear.
9:30	"	154	Impregnant boiling on surfaces. Runout on screen & in the pan.
10:05	"	158	Boiling has ceased. Resin is likely cured.
-(2) 10:40	"	156	Unload to desiccator. Set oven at 82°C (0.1%). Cool s/c compartment to weight.
-	-	-	$W_t = 1232.35g \Rightarrow W + Pickup = 93.51g \Rightarrow W/10 Pickup = 8.21$ (Yd = <u><u>51.6</u></u> %).
-	-	-	$\text{Pan + Screen + Resid} = 188.4g \Rightarrow \text{Cured Run-out} = \underline{\underline{7.0g}}$
* 11:55	82	245	Load over (180° Rotation * to bottom).
-(3) 13:55	"	248	Power off. Allow oven to cool to ~150°C \Rightarrow unload to desiccator for
15:25	OFF	156	Unload to desiccator. Cool overnight \Rightarrow weigh following morning

Post Curing Data: (5/18/01)

$$\text{Pan + Screen + Cured Run-out}_{(P)} = 188.3g \Rightarrow \text{Cured Run-out} = \underline{\underline{6.9g}}$$

$$W_{t,0} = 1212.74g \Rightarrow W + Pickup = 73.2g \Rightarrow W/10 Pickup = \underline{\underline{6.4g}} \quad (\text{Yd} = 40.8\%)$$

$$\Rightarrow \text{Impregnant Yield (including the runout)} = \underline{\underline{44.6\%}} \Rightarrow \text{Density}_{(L)} \approx \underline{\underline{1.620 g/cc}}$$

Label 195-129-56, Give to P. S. Rocco 5/21/01

Rebunker Portion: Ref. 195-129-76

Performed and Recorded by:

Directed by: J. C. Liu

Read and Understood by:

Date: 1/1

Date:

Date:

Material:

195-129-56: C/C composite via BP process from the 4th Block in the 1st Lawrenceburg trial. 0.25" long pitch fibers + Reitley 155 pitch. Load Ration 85/15 + 5 wt% sulfur based on pitch wt. Initial Data: Ref. 195-129-52 #4-1. VI & Cure Data: Ref. 195-129-55 & 56. Rec'd from P. Sirocky 6/20/01.

Rebake cycle (per P. Sirocky):

10°C/hr to 900°C, 2 hr hold. Block was warm when received. Cooled in desiccator \Rightarrow weighed & dimensioned.

Rebake Data: (6/20/01)

WT (g)	L ₁	L ₂	L ₃	AVE. LEN.	W ₁	W ₂	W ₃	AVE WIDTH	H ₁	H ₂	H ₃	Ave HT.	VOL (cc)
1129.43	192.10	192.32	192.37	192.36	83.98	83.62	82.86	83.49	46.55	46.42	46.60	46.59	742.836

Rebake Density = 1.577 g/cc $\Rightarrow \Delta = 0.055$ g/cc over "green" density.

Wt% (green thru rebake) = 3.56%

Vol% (green thru rebake) = -0.07% (essentially no change)

Delta (green thru rebake) = 3.61%.

Wt% (cure thru rebake) = -2.75%.

Returned to P. Sirocky 6/20/01. Data communicated via e-mail to D. Hoang, P. Sirocky, & E. Parast.

Comments:

* Impregnant % wt Yield from VF thru Rebake = $(40.59 / 181.27) \times 100 = 22.4\%$.

Note:

Mod MCC = 34.4 ($\sigma = 0.26, n = 3$) for the impregnant \Rightarrow The difference is due to run-out and/or weight loss from the pitch binder because it hadn't been to ~900°C yet.

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

Subject Initial Data of C/C Composites via BP Process (3rd Trial)

(C/C Comp)

Cross-Reference (if any)

Purpose:

To obtain initial weights and dimensions prior to vacuum impregnation with silicon (IV) oxide colloidal dispersion for insitu conversion to SIC.

Materials:

C/C composite via BP process. Rec'd from P. Strokey 7/16/01. Two brick sections in ~ half. From the 3rd Lawrenceburg trial. Made w/ 0.25" long K-223SE pitch fibers and Raileigh 155 pitch. Load ratio 75/25 w/o sulfur.

Procedure:

Ultrasonic washed in deionized H₂O 3x, 5 min intervals. \Rightarrow Vacuum dried on at ~166 °C, 0.1 mm pressure.

Air cooled, will not fit in desiccator, \Rightarrow weighed & dimensioned.

Initial Data: (7/19/01)

File Path = c:\Program Files\Excel\BP C-C Composites\Initial.xls Sheet = BP III

BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS

Material:

Material: BP-III-1 and BP-III-2. Rec'd 7/16/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/18/01. Dimensions were obtained with a Starrett No. 123-12 vernier caliper. Not vacuum dried at 166 °C to 0.1 mm pressure from 7/18 to 7/19/01. Weights obtained on Mettler PN 2210 balance on 7/19/01.

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
3-1-A	916.19	8.175	8.155	8.110	8.146	3.261	3.305	3.304	3.290	1.333	1.302	1.288	1.308	574.297	1.595
3-1-B	823.65	8.196	8.200	8.197	8.198	3.296	3.271	3.248	3.272	1.253	1.273	1.259	1.262	554.502	1.485
AVERAGE =															
STND. DEV. =															

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
3-2-A	867.13	8.883	8.863	8.837	8.861	3.226	3.265	3.298	3.263	1.183	1.170	1.169	1.174	556.247	1.559
3-2-B	861.58	8.913	8.905	8.896	8.905	3.263	3.281	3.292	3.279	1.196	1.207	1.180	1.194	571.400	1.508
AVERAGE =															
STND. DEV. =															

Dimensioned: 07/19/01
Not Vac. Dried: 07/18-19/01
Weighed: 07/19/01
N.B. Ref. No.: 195-129-86

#	Impregnant	NB Reference
3-1-A	Silicon (IV) Oxide Colloidal Dispersion	195-129-88+84
3-1-B	" " "	195-129-92+93
3-2-A	" " "	2VJ 195-129-94+95
3-2-B	" " "	195-129-96+97

Performed and Recorded by: 
 Directed by: 
 Read and Understood by:

Date
 Date
 Date

Subject: VI of 195-129-86 #3-1-A w/ SICer (IV) Oxide Colloidal Dispersion C/c/c com
Cross-Reference (if any)

Purpose:

To investigate insitu conversion of carbon to SiC in a c/c composite

Materials:

- 1) c/c Composite: 195-129-86 #3-1-A; (c/c composite via BP process from the 1st Blot of the 3rd Lawrenceburg trial. 0.25" long K-223SE pitch Fibers and Reiley 1055 pi. Load ratio 75/25. No sulfur. Wt_{c/c} = 916.19 g, Vol_{c/c} = 574.297 cc, Density = 1.595 g
- 2) Impregnant: Silicon (IV) Oxide, 30% in H₂O, colloidal dispersion. (Alfa-Aesar), lot # A04K09, 0.01 um particles, in liquid, S.A. = 320 m²/g. Density = Visc_{c/i} = 7.7 cps at 82.1°F, S.G._{c/i} = 1.216 at 82.1°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 + 16 Use teflon support and Pyrex pan for drying.

Pump Down Data: (7/20-23/01)

DATE	TIME	PRESS (inert)	Comments
7/20	12:45	18	Load c/c composite from cold, dry oven.
"	13:00	"	Begin pump-down.
"	14:00	44	
"	15:55	28	
7/23	7:20	21	
"	8:00	"	Charge traps w/ dry ice-acetone.
"	9:05	14	LDR
"	11:30	13	Begin VI

Impregnation Data: (7/23 + 24/01) - LDR w/traps charged

LDR: Init. Pres = 14 mTorr Visc_{c/i} = 7.7 cps at 82.1°F
 5 min = 27 " S.G._{c/i} = 1.216 at 82.1°F
 10 min = 38 "
 15 min = 49 "

Drop time = 11:50 (13 mTorr)

Unload time = 8:00 (7/24)

Held at atmospheric pres.
 for ~20 1/4 hrs.

Comments:

500 cc cylindrical funnel is full. Return impregnant to ② lqt. poly-bott

Post Impregnation Data: (7/24/01)

$$Wt_{(PVI.1)} = 1029.86 \text{ g} \Rightarrow Wt_{P.E. \text{ Pickup}} = 113.67 \text{ g} \Rightarrow Wt_{\text{Liquor Pickup}} = 12.41 \text{ g} \quad \text{Voll. Liquor Pickup} = 16.$$

Performed and Recorded by: J. J. Lin

Date: 7/24/01

Directed by: J. J. Lin

Date: 7/24/01

Read and Understood by:

Date: 7/24/01

Subject VI-195-129-86 #3-1-A wtsil. carIII on. ilp Collodial Dispersion (c/c compAP) 89
Cross-Reference (if any)

Drying Data: (7/24/25/01) - oven set "29" (50%). Argon purge = 5.0 scfm (41R)

TIME	OVEN TEMP	PRESS (mm)	Comments
* 8:10	108	Atm	Load oven; * to top. oven set "29" (50%). Purge w/gas at 5.0 scfm (41R)
(3) 11:10	106	"	Unload oven. Set oven at "38" (50%). Weigh brick hot (it doesn't fit in balance)
-	-	-	Wt = 989.02g \Rightarrow Wt. Pickup = 72.81g \Rightarrow Wt/o Pickup = <u>7.95</u> (Yd = <u>64.1%</u>)
* 11:30	125	Atm	Load oven; * to bottom. oven set at "38" (50%). Purge w/gas at 5.0 scfm (41R)
(2) 13:30	130	"	Unload oven. Set oven at "46" (50%). Weigh brick hot. Install new gasketing.
-	-	-	Wt = 954.41g \Rightarrow Wt. Pickup = 38.22g \Rightarrow Wt/o Pickup = <u>4.17</u> (Yd = <u>33.4%</u>)
* 14:20	126 (n)	736.6	Load oven; * to top. oven set "46" (50%). Vac. pump on. Argon purge off. Reduce pressure.
(7) 7:20	175	0.4	Vac. pump off. Pressure rise w/gas. Set oven at "29" (50%). Leave door open to cool.
-	-	-	Wt = 952.26g \Rightarrow Wt. Pickup = 36.07g \Rightarrow Wt/o Pickup = <u>3.94</u> (Yd = <u>31.7%</u>)

Comments:

1) After 3 hrs, $\sim 107^\circ\text{C}$, atm pressure;

No evidence of run-out. Set oven at "38" (50%). Rotate brick 180° (* to bottom).

2) After 2 hrs, $\sim 128^\circ\text{C}$, atm pressure;

No evidence of run-out. Set oven at "46" (50%). Rotate brick 180° (* to top). Remove old door gasketing and replace w/new gasketing

3) After 17 hrs, $\sim 175^\circ\text{C}$, vacuum

Note: by 15:30 (7/24/01) the oven temperature was $\sim 160^\circ\text{C}$ and the pressure was 0.1mm \Rightarrow sample dried quickly.

No evidence of run-out. Set oven to "29" (50%) and leave door open to cool.

Comments:

The percent weight yield of the impregnant, *in situ*, agrees w/that of the solution in crucible. Ref. 195-129-90.

Label 195-129-89

Performed and Recorded by:

Directed by: J. Cen

Read and Understood by:

Date - / /

Date - / /

Date - / /

Subject: Drying of ~10g samples of silicon (IV) oxide, 30% in H₂O, colloidal dispersion (decomp)
Cross-Reference (if any)

Purpose:

To determine the percent yield by weight of solution to compare w/ insitu percent weight yield of Vfcl c/c composites.

Material:

silicon (IV) oxide, 30% in H₂O, colloidal dispersion (AlF₃-Aesop), 40+ A04K09,
0.01 um particles, in liquid. SA = 320 m²/g. Density = 1.20.
 $V_{B(I)} = \frac{7.7}{\text{c.p.s.}} \text{ at } 82.1^{\circ}\text{F}$, Spec. Grav._{s,I} = 1.216 at 82.1 °F.

Apparatus:

② 100 ml porcelain. Al foil covers.

Procedure:

Weigh ~10g into each crucible, containing SiC boiling chips. Cover w/ Al foil.
 Punch holes in Al foil and obtain total weight. Subtract sample weight
 to obtain TARE. Processed w/ 195-129-89.

Initial Data: (7/24/01)

$$\begin{aligned} 1) \text{CRUC CHIPS + SAMPLE (I)} &= 59.4475 (\dagger) \quad \text{CRUC (etc.) + Al Foil (I)} = 60.1600 \\ \text{CRUC + CHIPS (I)} &= 49.3229 \quad \text{CRUC (etc.) (I)} = \underline{59.4475} \\ \text{SAMPLE (I)} &= \underline{10.1746g} \quad \text{Al Foil (I)} = 0.6620g \Rightarrow \text{TARE} = \underline{49.4854} \end{aligned}$$

$$\begin{aligned} 2) \text{CRUC + CHIPS + SAMPLE (II)} &= 54.7967 (\dagger) \quad \text{CRUC (etc.) + Al Foil (II)} = 55.4580 \\ \text{CRUC + CHIPS (II)} &= 44.7620 \quad \text{CRUC (etc.) (II)} = \underline{54.7967} \\ \text{SAMPLE (II)} &= \underline{10.0347g} \quad \text{Al Foil (II)} = 0.6613 \Rightarrow \text{TARE} = \underline{45.4233} \end{aligned}$$

Drying Data: (7/24+25/01) - Processed w/ 195-129-89

	TIME	OVEN TEMP	PRES (mm)	Comments
*	8:10	108	Atm	Load in oven; in front of Pyrex tray w/ 195-129-89. Purge w/larger at 5.0 scFH(Gas)
(3)	11:10	106	"	Unload, cool in desiccator \Rightarrow weigh. Set over at "38" (50%).
-	-	-	"	#1 Cruc (etc) Wt = 54.7662g \Rightarrow Wt = 4.7808g \Rightarrow Wt Yield = <u>46.99%</u>
-	-	-	"	#2 Cruc (etc) Wt = 49.9430g \Rightarrow Wt = 4.5197g \Rightarrow Wt Yield = <u>45.04%</u>
*	11:30	125	Atm	Reload; switch sides. Over set "38" (50%). Purge w/larger at 5.0 scFH(Gas)
(2)	13:30	130	"	Unload, cool in desiccator \Rightarrow weigh. Set over at "46" (50%).
-	-	-	"	#1 Cruc (etc) Wt = 53.2721g \Rightarrow Wt = 3.2867g \Rightarrow Wt Yield = <u>32.30%</u> .
-	-	-	"	#2 Cruc (etc) Wt = 48.6710g \Rightarrow Wt = 3.2477g \Rightarrow Wt Yield = <u>32.36%</u> .
				cont'd next page

Performed and Recorded by: J. Lew

Date

Directed by: J. Lew

Date

Read and Understood by:

Date

Subject Drying of ~10g samples of S.I. iron (II) Oxide, 3w% in H₂O, colloidal dispersion (etc contd B.P.) 91
Cross-Reference (if any)

Drying Data (cont'd): (7/24 + 25/61)

TIME	OVEN TEMP (°F)	PRESS (mm)	Comments
* 14:20	126 (m)	736.6	Reheat; reverse sides. Or use set "40" OVEN. Vac. pump off. Argon purge off.
• 07	175	0.4	Vac. pump off. Pressurize stronger. Unload, cool in desiccator. → weight.
-	-	-	(1) (calc'd) $W_t = 53.2182g \Rightarrow W_t = 3.2328g \Rightarrow W_t Y_{yield} = 31.77\%$
-	-	-	(2) (calc'd) $W_t = 48.6127g \Rightarrow W_t = 3.1894g \Rightarrow W_t Y_{yield} = 31.78\%$

Ave % wt yield = 31.8, $\sigma = 0.01$, $n = 2$

Comments:

Almost exactly the same value for each sample. Same percent weight yield as the insitu i. wt.yield for 195-127-89.

Performed and Recorded by:

Directed by: *J. Liu*

Read and Understood by:

Date

Date

Date

Subject: VI-F 195-129-86 #3-1-B w/ silicon (IV) oxide, Colloidal Dispersion (c/c com
Cross-Reference (if any)

Purpose:

Ref. 195-129-88

Materials:

- 1) c/c Composite; 195-129-86 #3-1-B; c/c composite via BP process from the 1st block of the 3rd Lawrenceburg trial. 0.25" long K-223-SF pitch fibers and Reiley 105, load ratio 75/25. No sulfur. Wt (2) = 823.65g, Vol (2) = 554.502cc, Dens(2) = 1.485g/l
- 2) Impregnant: silicon (IV) oxide, 30% in H₂O, colloidal dispersion. (AIFa-Aesar), lot # A04K09 0.01 um particles, in liquid. SA = 320 m²/g. Dens. 2g = 1.20. Previous use is 195-129-8 V_{1.3}(2) = 7.7 cps at 82.1°F, S.G.(2) = 1.216 at 82.1°F.

Apparatus:

Ref. 195-120-105

Procedure:

Ref. 195-129-88

Pump-Down Data:

DATE	TIME	PRESS (mTorr)	Comments
7/23	13:15	21	Load block from hot oven (~110°C, atm)
"	13:25	"	Begin pump-down
"	14:25	45	
7/24	7:10	24	
"	8:25	23	charge traps w/dry ice-acetone
"	8:55	15	LDR
"	11:45	13	Begin VE

Impregnation Data: (7/23 & 24/01) - LDR w/traps charged.

LDR: Initial = 14 mTorr Vis = 7.8 cps at 78.0°F Drop Time = 11:45 - (13 mTorr)
 5 min = 27 " 5.6. = (1.216) at 78.0°F Unload Time = 7:45 (7/25/01)
 10 min = 38 " Held at atmospheric pressure for
 15 min = 49 " ~20 hrs.

Comments:

500 ml cylindrical funnel filled (ie - ~675 ml)

Post Impregnation Data: (7/25/01)

$$Wt_{(PV1-1)} = 950.80g \Rightarrow Wt_{\text{Pickup}} = 127.15g \Rightarrow Wt_{\text{to Pickup}} = (15.44) \text{ Vol to Pickup} = 18.86$$

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

Subject V.F. # 125-128-86 #3-1-B w/size/cm (xx)oxide, colloidal dispersion (col comp BP) 93
Cross-Reference (if any)

Drying Data: (7/25 + 26/01) - oven set "29" (50°C). Purge after open + 5.0 SCFH(AIR)

	TIME	OVEN TEMP (°C)	PRESS (mm)	Comments
*	8:00	107	Atm	Load oven; # to top. Over set "24" (50%). Argon purge 5.0 scfm (Air)
(3)	11:00	108	"	Unload oven. Over set "38" (50%). Weigh block hot.
-	-	-		$W_t = 907.62 \text{ g} \Rightarrow W_t / \text{Pickup} = 83.97 \text{ g} \Rightarrow W_t / \text{Pickup} = 10.19 \text{ (Yd = 66.0\%)} \text{ (1)}$
*	11:20	129	Atm	Load oven; # to bottom. Over set "38" (50%). Argon purge 5.0 scfm (Air)
(2)	13:20	132	"	Unload oven. Over set "46" (50%). Weigh block hot. Install new gasketing.
-	-	-		$W_t = 871.76 \text{ g} \Rightarrow W_t / \text{Pickup} = 48.11 \text{ g} \Rightarrow W_t / \text{Pickup} = 5.84 \text{ (Yd = 37.8\%)} \text{ (2)}$
*	13:40	134 (4)	736.9	Load oven; # to top. Over set "46" (50%), Vac. pump on. Argon purge off.
(8)	7:40	168	0.4	Vac. pump off. Pressure write warning. Set oven at "29" (50%).
-	-	-		$W_t = 863.89 \text{ g} \Rightarrow W_t / \text{Pickup} = 40.34 \text{ g} \Rightarrow W_t / \text{Pickup} = 4.89 \text{ (Yd = 31.6\%)} \text{ (3)}$

Comments:

- 1) After 3 hrs, $\sim 108^\circ\text{C}$, atm. pressure;
 Rotate brick 180° (# to bottom). Set oven at $"38"$ (50%). No evidence of run-out.
 Similar % wet yield to 195-129-89 (i.e. 64.1%).
- 2) After 2 hrs, $\sim 131^\circ\text{C}$, atm pressure;
 Scrape off old gasketing and install new. Set oven to $"46"$ (50%). Rotate
 brick 180° (# to top). No evidence of run-out. H₂C condensed on oven door;
 wiped dry.
- 3) After 18 hrs, $\sim 168^\circ\text{C}$, vacuum;
 No evidence of run-out. Oven set to $"29"$ (50%). Leave door open to cool.

Label 195-129-93

Graph Doctor: Ref. 195-130-63

Performed and Recorded by:
Directed by: *J. C. L.*
Read and Understood by:

Date / /
Date / /
Date / /

94 Subject: ~~1-1~~ 195-129-86 3-2-A w/silicon(IV)oxide, colloidal dispersion (etc cont.)
Cross-Reference (if any)

Purpose:

Ref. 195-124-88

Materials:

1) CIC Compos. Re 1917-1AS-86 #3-2-A Let's composite via BP process. From block #2 of the 3rd Lawrenceburg trial. 0.25" long K-223SE pitch fibers. Reilly 105 pitch. Load Ratio = 75/1
wt% sulfur. Wt_{CO} = 867.13g, Vol_{CO} = 556.247cc, Dens_{CO} = 1.1557g/cc

2) Impregnated silica (II) oxide, 30% in H₂O, colloidal dispersion. (Alfa-Aesar), Lot # A004
 0.01um particles, in liquid. SA = 320 m²/g. Density = 1.20, Previous use: 7/25/01.
 Viscos: 747 cps at 82.1°F, 56cps: 1.316 at 82.1°F.

نذر ملکہ عزیزہ

Ref. 195-130-45

Precursors

Left. 195-129-15 + 16 * Processed w/ 195-129-86 #3-2-13

Pump Down Date: (7/24/2011)

DATE	TIME	MEAS (cm)	Comments
7/24	13:20	19	Load from over lab
"	13:35	"	Began pump - down
"	14:35	340	"High" pressure \Rightarrow bridle picked up moisture in lab
"	15:35	~105	
7/25	7:20	37	
"	8:35	33(4)	Change traps w/ dry ice - acetone
"	9:20	22	LDR
"	11:45	18	Reopen VIT

Ingraham Data: (7/25 & 26/01) - LOB vs traps charged

LDR: Initial = 22 mTorr V_{CE} = 7.4 Vps at 78.0°F

$$T_{\text{max}} = 76 \quad " \quad S_{G, 17.5} = 1.330 / \text{at } 76.0^{\circ}\text{F}$$

10-12-56 100% - 100% - 100%

10 min : 60 "

$$15 \text{ min} = 74 \quad \text{a}$$

Drop Time = 11:45 (16 mTc)

$$13.3 \text{ focal length} = 8.115 \text{ cm}$$

Held at atmospheric pressure
for $\sim 20\frac{1}{2}$ hrs.

Gennaro I.

Filled second cylindrical frame.

Post-Impragnation Data: (7/26/01)

$$w_{\text{t}}(\text{PvF-1}) = 987.35 \text{ g} \Rightarrow w_{\text{t}} \text{ Pickop} = 120.32 \text{ g} \Rightarrow w_{\text{el}} \text{ or } \text{Pckwgr} = 13.86, \text{ Volumen Pckwgr} = 17.$$

Performed and Recorded by:

Directed by: John

Read and Understood by:

Date

Bato
Date

Date _____

Subject: VI of 195-129-86 #3-2-A w/silicon nitride, colloidal dispersion (ct compo) 95
 Cross-Reference (if any)

Drying Data: (7/26+27/61) - Over set "29" (50%). Purge w/argon at 5.0 SCFH (AIR)

	TIME	OVEN TEMP (°C)	PRESS (mm)	Comments
*	8:30	106	Atm	Loud over; # to top. over set "29" (50%). Argon purge at 5.0 SCFH (AIR)
- (1)	11:30	120	"	Unloud over. over set "38" (50%). Weigh block hot.
-	-	-	Wt = 944.48g \Rightarrow Wt Pickup = 77.35g \Rightarrow Wt/o Pickup = 8.92 ($Y_d = 64.3\%$).	
*	11:45	130	Atm	loud over; # to bottom, front of tray. over set "38" (50%). Argon purge 5.0 SCFH
- (2)	13:45	138	"	Unloud over. over set "46" (50%). Weigh block hot. Install new gasketing.
-	-	-	Wt = 906.75g \Rightarrow Wt Pickup = 39.62g \Rightarrow Wt/o Pickup = 4.57 ($Y_d = 33.0\%$).	
*	14:30	176	742.0	Loud over; # to top, back of tray. Vac. pump off. Argon purge off. Reduce pressure.
- (18)	8:30	166	0.3	Vac. pump off. Pressurize w/argon. Set over at "29" (50%).
-	-	-	Wt = 905.02g \Rightarrow Wt Pickup = 37.89g \Rightarrow Wt/o Pickup = 4.37 ($Y_d = 31.5\%$.)	

Comments:

1) After 3 hrs, $\sim 113^{\circ}\text{C}$, atm. pressure;
 No evidence of run-out. Set over at "38" (50%). Weigh block hot. Rotate 180°;
 # to bottom. Reverse position w/ 195-129-86 #3-2-B (i.e. front of Pyrex tray).

2) After 2 hrs, $\sim 148^{\circ}\text{C}$, atm pressure;
 No evidence of run-out. Set over at "46" (50%). Weigh block hot. Rotate 180°; # to top. Reverse position w/ "B" (i.e. rear of Pyrex tray). Remove old gasketing and install new.

3) After 18 hrs, $\sim 171^{\circ}\text{C}$, vacuum
 Set over at "29" (50%). Weigh block hot \Rightarrow reload w/ "B" comparison into VI unit for 2nd impregnation

Comments:

3. yield of the impregnant agrees w/ previous ② blocks; 31.7%,
 31.6%, and 31.5%.

Label 195-129-95 #3-2-A $\xrightarrow{\text{ct}}$ VI: Ref. 195-129-99+100

Performed and Recorded by:

Directed by: J. Chin

Read and Understood by:

Date

Date

Date

Subject: VI-F 195-129-86 #3-2-B wts: heat (II) oxide, colloidal dispersion (etc comp.)
Cross-Reference (if any)

Purpose:

Ref. 195-129-88

Materials:

(1) c/c composite 195-129-86 #3-2-B (c/c composite via B/P process. From block #2 of the 3rd Lawrenceburg trial, 0.25" long K-223-16 pitch fibers. Rat/leg 15% p. etc). Load ratio = 75/12
wt% solvent. Wt% - 861.58g, Vol% = 531.400cc, Dens. = 1.508g/cc

(2) Impregnant: Silicon (II) oxide, 30% in H₂O, colloidal dispersion. (A) Fm-Aesar, lot # A01
0.01um particles, in liquid. SA = 320 m²/g. Density = 1.20. Previous use: 7/26/01.
Viscosity = 7.7 cps at 82.1°F, S.G. = 1.216 at 82.1°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15+16 * Processed w/ 195-129-86 #3-2-A

Pump-down Data: (7/24/02/01)

DATE	TIME	PRESS (in Torr)	Comments
7/24	13:20	19	Load from open lab w/ 3-2-A
"	13:35	"	Begin pump-down.
"	14:35	370	" High pressure → blocks picked up moisture in lab.
"	15:55	200	
7/25	7:20	370	
"	8:35	33(0)	Charge traps w/ dry ice-acetone
"	9:20	22	LDR
"	11:45	18	Begin VI

Impregnation Data: (7/25+26/01) - LDR w/ traps charged

LDR: Initial = 22 mTorr Vis. = 7.4 cps at 78.0°F Drop Time = 11:45 (18 mTorr)
 5min = 46 " SG = 1.216 (1.220) at 78.0°F Onset Time = 8:15 (7/26/01)
 10min = 60 " Dmin = 74 "

Comments:

Filled 500ml cylindrical funnel (i.e. v. 25ml).

Post Impregnation Data: (7/26/01)

Wt (PyI-1) = 945.39g → Wt Pickup = 133.81g ⇒ w/w Pickup = (15.53), Vol/o Pickup = 18.20

Performed and Recorded by: J. ...

Date:

Directed by: J. ...

Date

Read and Understood by:

Date

Subject VI #195-129-86 #3-2-B w/5.1% (III) oxidized, colloidal dispersion (check CHPBA)⁹⁷
 Cross-Reference (if any)

Day 25 Data: (7/26 + 27/61) - Over set "29" (50%). Argon purge 5.0 SCFH (4 hr). Process w/195-129-95

	TIME	OVEN TEMP.	PRESS (mm)	Comments
*	8:30	106	Atm	Load oven; # to top. Over set "29" (50%). Argon purge at 5.0 SCFH (4 hr).
(3)	11:30	120	"	Unload oven. Set oven "38" (50%). Weigh block hot.
-	-	-		Wt = 950.91g \Rightarrow Wt Pickup = 89.33g \Rightarrow Wt/o Pickup = 10.37 (Yd = 66.8%)
*	11:45	150	Atm	Load oven; # to bottom, back of tray. Over set "38" (50%). Argon purge 5.0 SCFH.
(2)	13:45	138	"	Unload oven. Set oven "46" (50%). Weigh block hot. Install new gasketing.
-	-	-		Wt = 906.40g \Rightarrow Wt Pickup = 44.90g \Rightarrow Wt/o Pickup = 5.21 (Yd = 33.6%)
*	14:30	176	742.0	Load oven; # to top, front of tray. Over set "46" (50%). Vac. pump on. Argon purge off.
(6)	8:30	166	0.3	Vac. pump off. Pressureize w/argon. Set oven "29" (50%).
-	-	-		Wt = 903.5g \Rightarrow Wt Pickup = 43.01g \Rightarrow Wt/o Pickup = 4.86 (Yd = 31.4%)

Comments:

1) After 3 hrs, ~1/3°C, atm pressure;

No evidence of run-out. Set oven at "38" (50%). Weigh block hot. Rotate 180°; # to bottom. Reverse position in Pyrex tray w/195-129-86 #3-2-A (i.e. back of tray).

2) After 2 hrs, ~144°C, atm pressure;

No evidence of run-out. Set oven at "46" (50%). Weigh block hot. Rotate 180°; # to top. Reverse position in Pyrex tray (i.e. front of tray). Remove old door gasketing and install new.

3) After 18 hrs, ~171°C, vacuum;

Set oven "29" (50%). Weigh block hot \Rightarrow reload w/195-129-95 #3-2-A into VI unit for 2nd impregnation.

Comments:

1. yield of impregnant agrees w/ previous ③ blocks; 31.7%, 31.6%, and 31.5%.

Label 195-129-97 #3-2-B 2nd VI: Ref. 195-130-01202

Performed and Recorded by:

Directed by: J. Am

Read and Understood by:

Date 5/11/

Date

Date

Subject Initial Data of Graphitized c/c Composites via BP Process (4th Trial) (6k
Cross-Reference (if any)

Purpose:

To obtain the initial weights and dimensions prior to vacuum impregnation with "T-143" type phenolic/furfural resin blend for densification.

Materials:

c/c composites via BP process. Rec'd from R. Sirocky 7/25/01. Two sections, both graphitized. Section "A-1" had one pitch impregnation, section "B-1" did not have a PI. Both graphitized to ~3000°C

Made w/ K-2235E 0.25" long fibers and Reillyg 155 pitch. Load Ratio = 75/2 w/o softwr. Brick 13 of 4th trial.

Procedure:

Essentially same as 195-129-86. Except coated in desiccator

Initial Data! (7/27/01)

File Path = C:\Program Files\Excel\BP c/c composites\In.Trial.xls Sheet = BP II

BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS

Material:

Material: BP-IV-13 A1 and BP-IV-13 B1. Rec'd 7/25/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/26/01. Dimensions were obtained with a Mitutoyo Model CD-8-CS digital caliper. Hot vacuum dried at 124 °C to 0.4 mm pressure from 7/26 to 7/27/01. Weights obtained on Mettler BN 2210 balance on 7/27/01.

Note: Both samples have been graphitized to ~3000°C. A1 has one PI. B1 has no PI.

Sample I.D.	Weight (g)	L1 (mm)	L2 (mm)	L3 (mm)	Ave. Length (mm)	W1 (mm)	W2 (mm)	W3 (mm)	Ave. Width (mm)	H1 (mm)	H2 (mm)	H3 (mm)	Ave. Height (mm)	Vol. (cc)	Dens. (g/cc)
4-13-A1	277.03	108.95	108.89	108.98	108.94	93.90	93.92	93.95	93.92	15.65	16.01	16.08	15.91	162.825	1.701
4-13-B1	255.92	107.20	106.19	105.13	106.17	96.81	97.06	97.50	97.12	16.48	16.15	15.66	16.10	165.987	1.542

Dimensioned: 07/27/01

Hot Vac. Dried: 07/26-27/01

Weighed: 07/27/01

N.B. Ref. No. : 195-129-98

Impregnant

N.B Ref.

4-13-A1 195-129-53 (w/50 by vol to G.P.S+32/Furfural) 195-130-03 ± 04
4-13-B1 " " " " " / " 195-130-05 ± 06

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date